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The European Patent Office  
PB5818 Patentlaan 2  
NL-2280 HV Rijswijk  
Netherlands

Dear Sirs

Re: PCT Patent Application No. PCT/GB2004/001373  
(Filed 30th March 2004) based on  
UK Patent Application No. 03 07290.7  
(filed 31st March 2003)  
Applicant: Middlesex Silver Co. Limited  
(assigned from COLE, Paul Gilbert)  
Inventors: JOHNS, Peter Gamon and HARRISON, Clare Elizabeth  
"Enhancing Silver Tarnish-Resistance"  
**Case: MDX,001-PCT**

FAO: International Section

In response to the first written opinion we file herewith new description pages 9-11 and new claims pages 36-38. We also file herewith a copy of the claims printed using the TRACK CHANGES tool of WORD, showing the changes that have been introduced from the claims as filed.

PLEASE NOTE THAT A FAVOURABLE INTERNATIONAL PRELIMINARY EXAMINATION REPORT IS IMPORTANT TO THESE APPLICANTS. IT IS THEREFORE REQUESTED THAT A FURTHER WRITTEN OPINION SHOULD BE ISSUED, OR THAT THE EXAMINER SHOULD TELEPHONE THE UNDERSIGNED REPRESENTATIVE, IF IT IS NOT POSSIBLE TO REPORT FAVOURABLY ON THE CLAIMS AS AMENDED.

The claims as amended now all refer to the treatment of finished or semi-finished flatware, hollowware or jewellery articles (i.e. articles of the kind made by silversmiths) and now all specify silver and germanium contents for the articles being treated. They also all specify that the sample of the alloy when supported close above a 20% solution of ammonium polysulphide for at least 30 minutes retains a generally untarnished appearance. As explained in applicant's specification, that is a much higher degree of tarnish resistance than is obtainable for conventional Sterling silver.

contn:-

As acknowledged by the Examiner, treatment of the present alloys with an alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide is novel having regard to the disclosure of D1.

The examiner cites D2 to show that it was known that alkane-thiols would form layers on the surface of germanium. However, as explained in the Background section of applicant's specification, D2 relates to semiconductor and nanotechnology, and documents in this remote field would be most unlikely to be consulted by silversmiths. D2 was found in the course of searches made with hindsight knowledge of the invention, and the true position is as set out at page 2 lines 4 and 5 - i.e. that the literature on formation of alkylthiols of germanium is sparse and unhelpful except in impermissible hindsight.

Furthermore, even if a skilled person were to read the disclosures of D1 and D2 together, he would note that germanium is contained in the present alloys in relatively small amounts, There is nothing to suggest that the present very high levels of tarnish resistance can be achieved with the present alloys when they are not obtained with standard Sterling silver, and still less that the thiol film is durable and effective in the case of the present alloys whereas it is less effective and removable by an n-propyl bromide based solvent in the case of standard Sterling silver. The achievement of a degree of tarnish resistance higher than that obtainable with standard Sterling is a technical feature of the method of claim 1 as amended and should be taken into account when assessing inventive step.

It is noted that the subject matter of claims 5 and 6 is considered to meet the requirements of the PCT.

Claim 7 as amended now specifies that said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in a composition obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide direct in an aqueous mixture of an anionic surfactant and a neutral, amphoteric or zwitterionic surfactant, said mixture being free from solvents other than water. The references discussed at pages 5-7 show that finding suitable solvents for these materials was difficult and that a number of attempts had been made over a period of many years. The discovery that it is possible to disperse the materials directly in aqueous liquids containing an anionic and a neutral or amphoteric surfactant and free from all other solvents is surprising and advantageous given the increasing difficulty and expense of using solvent-based systems. The combination of surfactants of claim 8 has been found to be particularly effective.

Claim 19 specifies that the treated article is introduced into packaging, and refers to the problem referred to in the para-

contn:-

graph beginning at page 7 line 23 of tarnishing during display in a retail environment. If a manufacturer surface treats articles of the present alloy and then packs them for delivery to a retailer, the article when unpacked and displayed by the retailer should remain untarnished until it is sold. It is submitted that this is not only a technical effect but also a result of practical importance because it greatly reduces the effort and cost needed to sell silverware.

The description has been adapted to the claims as amended.

We await the result of the further examination of this application.

We enclose the Demand for ALL countries originally designated including the necessary Fee Calculation Sheet totalling EUR 1659 to be deducted from our deposit account no. 28050111.

Would you please return the attached copy of this letter to acknowledge safe receipt hereof.

Yours faithfully

// *Paul Cole*

Paul Cole/jn  
Chartered Patent Agent  
European Patent Attorney

Enc: Description pages  
New claims  
Track changes doc  
Demand

Records:-

Status - 13 Nov 04

lighting, an article of conventional Sterling silver would require re-polishing after one week and after two weeks would normally be so tarnished as to be unsaleable. At an exhibition, the life of an article on display before significant tarnish sets in may be as short as 3-4 days. Re-polishing produces wear and fine  
5 handling scratches, so that unless the article can be sold quickly it loses its pristine appearance. The need to polish display silver at frequent intervals adds to the labour cost of a jeweller or other retail establishment, whose management take the view that its staff should be employed to sell products and not to clean stock. Tarnish at point of sale or display is therefore a serious problem that reduces the  
10 willingness of those in the distribution chain to stock and display silver products, and which has not yet been adequately solved.

When the product reaches the ultimate purchaser, it is of course desirable that the task of tarnish removal should be made as infrequent and undemanding as  
15 possible.

Silver alloys according to the teaching of GB-B-2255348 and EP-B-0729398 are now commercially available in Europe and in the USA under the trade mark Argentium, and the word "Argentium" as used herein refers to these  
20 alloys. Although they exhibit improved tarnish resistance compared to e.g. Sterling silver, and any tarnish that forms can be removed by simple washing, there is still room for improvement in tarnish resistance. That remains true even when annealing is conducted in a selectively oxidising atmosphere as disclosed in WO 02/095082.

25

It has now been found that an alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide can be used for the surface treatment of an alloy of silver containing an amount of germanium that is effective to reduce firestain and/or tarnishing so as to reduce or further reduce tarnishing of the alloy such that  
30 a sample can be subjected to hydrogen sulphide gas above a 20% solution of

ammonium polysulphide for at least 30 minutes and typically 45-60 minutes at room temperature while retaining a generally untarnished appearance.

The invention therefore relates to a method for treating a finished or semi-  
5 finished shaped flatware, hollowware or jewellery article of a silver/germanium alloy that has a silver content of at least 77 wt % and a germanium content of between 0.4 and 7% the remainder principally being copper so as to reduce or further reduce tarnishing of the article such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium  
10 polysulphide for at least 30 minutes while retaining a generally untarnished appearance, said method comprising

surface treating said article with an alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide

15 The above method may include the further step of introducing the article into packaging.

The invention further provides a finished or semi-finished shaped flatware, hollowware or jewellery article of an alloy of silver containing an  
20 amount of germanium that is effective to reduce firestain and/or tarnishing and that has been treated with a C<sub>12</sub>-C<sub>24</sub> alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide and that exhibits a tarnish resistance such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a  
25 generally untarnished appearance.

The above accelerated tarnish test in which the article is subject to hydrogen sulphide gas from the ammonium polysulphide solution above which it is suspended at a height of e.g. 30mm corresponds to a period of a year or more in  
30 a retail environment where an article is on display and exposed to ambient atmosphere and may be subject to elevated temperatures. It is the combination of

the protective function of the germanium content of the alloy with the further protection from the organo-sulphur compound that is believed to be responsible for the observed increase in tarnish resistance. The period during which the article retains its untarnished appearance under these severe conditions may be three or  
5 more times the corresponding period for an article that has not been treated with an organo-sulphur compound, which is unexpected because the same accelerated tarnish test carried out under the same conditions on a conventional Sterling silver article not containing protective germanium does not reveal a significant increase in untarnished lifetime between its untreated and organo-sulfur treated states.  
10 Accelerated tarnishing trials carried out using Argentium and standard Sterling silver samples immersed in solutions of octadecyl mercaptan and hexadecyl mercaptan have shown that the protective thiol is removed from the standard Sterling sample but not from the Argentium silver samples on rubbing with a tissue soaked in a solvent (EnSolv 765, an n-propyl bromide based solvent  
15 cleaner discussed below). In accelerated testing the solvent-rubbed regions of standard Sterling silver discolour more rapidly than the un-rubbed regions whereas in Argentium silver no noticeable difference in appearance develops between the rubbed and un-rubbed regions, suggesting that thiol bonding is stronger or more effective.

20

Accelerated tarnishing tests with Argentium Sterling using ammonium polysulphide have been reported by the Society of American Silversmiths, see <http://www.silversmithing.com/1argentium4.htm>  
and in a comparative test the Argentium Sterling remained untarnished after one  
25 hour whereas conventional Sterling became tarnished after less than 15 minutes. However, in this test 0.5ml of 20% ammonium polysulfide solution is mixed with 200ml of distilled water, so that the test is greatly less severe than when samples are exposed to the 20% solution itself. In WO 02/095082, samples were suspended above 20% ammonium polysulphide, but the exposure times were  
30 relatively short, and onset of yellowing was reported for Ag-Cu-Ge alloys after 3-5 minutes exposure. Other tests reported in that specification involve placing

samples in a desiccator containing flowers of sulphur and calcium nitrate and are less severe than the ammonium polysulphide test.

5       As part of their program for developing improved formulations for the  
treatment agents described above, the applicants have unexpectedly discovered  
that the treatment agents can be dissolved or dispersed directly in aqueous  
surfactant without the need for preliminary dissolving of the treatment agent in an  
organic solvent and subsequent mixing of the resulting solution with aqueous  
liquid. Embodiments of the above compositions are optically clear and storage-  
10       stable at ambient temperatures for a period of weeks or months. The treatment  
composition may therefore be water-based and comprise an alkanethiol, alkyl  
thioglycollate, dialkyl sulfide or dialkyl disulfide and a mixture of an anionic  
surfactant with a neutral or amphoteric surfactant and water.

15

## CLAIMS

1. A method for treating a finished or semi-finished shaped flatware, hollowware or jewellery article of a silver/germanium alloy that has a silver  
5 content of at least 77 wt % and a germanium content of between 0.4 and 7% the remainder principally being copper so as to reduce or further reduce tarnishing of the article such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance, said method  
10 comprising  
surface treating said article with an alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide
2. The method of claim 1, wherein the alkanethiol, alkyl thioglycollate,  
15 dialkyl sulfide or dialkyl disulfide has C<sub>12</sub>-C<sub>24</sub> alkyl groups.
3. The method of claim 1 or 2, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in an organic solvent.
- 20 4. The method of claim 3, wherein the solvent containing the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is generally neutral.
5. The method of claim 3 or 4, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in a solvent based on n-propyl bromide.  
25
6. The method of claim 3 or 4, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in (a) a composition obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in an organic solvent and adding to said solution a relatively concentrated aqueous soap  
30 or detergent, or (b) an aqueous dispersion obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in an organic



solvent, adding to said solution a relatively concentrated aqueous soap or detergent, and diluting the resulting mixture with water.

5 7. The method of claim 1 or 2, wherein said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in a composition obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide direct in an aqueous mixture of an anionic surfactant and a neutral, amphoteric or zwitterionic surfactant, said mixture being free from solvents other than water.

10 8. The method of claim 7, wherein said composition comprises as surfactant a betaine.

9. The method of claim 8, wherein the betaine is cocamidopropyl betaine.

15 10. The method of any of claims 7-9, further comprising an anionic surfactant.

11. The method of claim 10, wherein the anionic surfactant is of the formula  $RO-(CH_2CH_2)_nSO_3M$  wherein R represents  $C_{10}-C_{18}$  alkyl, n is 2-6 and M represents a monovalent cation.

20

12. The method of claim 11, wherein the anionic surfactant is a monovalent cation salt of laureth sulfate.

13. The composition of any of claims 7-12, comprising amphoteric or  
25 zwitterionic surfactant and anionic surfactant in a weight ratio of from 1:10 to 10:1.

14. The method of claim 9, wherein the aqueous mixture comprises sodium laureth sulfate and cocamidopropyl betaine.

30

15. The method of any preceding claim, wherein the alkanethiol or alkylthioglycolate is selected from stearyl mercaptan (octadecyl mercaptan), cetyl mercaptan (hexadecyl mercaptan), stearyl thioglycollate and cetyl thioglycollate.
- 5 16. The method of any preceding claim, wherein the alloy consists, apart from impurities and grain refiner, of 92.5-98% silver, 0.3-3% germanium, and 1-7.2% copper, by weight of the alloy, together with 1-40 ppm boron as grain refiner.
- 10 17. The method of claim 16, wherein the ternary alloy consists, apart from impurities and grain refiner, of 92.5-96% silver, 0.5-2% germanium, and 1-7% copper, by weight of the alloy, together with 1-40 ppm boron as grain refiner.
18. The method of any of claims 1-15, wherein the alloy is a quaternary alloy of silver, copper, zinc and germanium.
- 15 19. The method of any preceding claim, comprising the further step of introducing the treated article into packaging.
- 20 20. The method of claim 19, wherein said packaging includes a presentation box.
21. The method of claim 20, wherein the packaging includes external wrapping for the presentation box.
- 25 22. Use of a C<sub>12</sub>-C<sub>24</sub> alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in the preparation of a tarnish inhibitor for an article of a silver/germanium alloy that has a silver content of at least 77 wt % and a germanium content of between 0.4 and 7%, the remainder principally being copper, so as to reduce tarnishing of the alloy such that a sample can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance.
- 30

23. A finished or semi-finished shaped flatware, hollowware or jewellery article of an alloy of silver containing an amount of germanium that is effective to reduce firestain and/or tarnishing and that has been treated with a C<sub>12</sub>-C<sub>24</sub> alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide and that exhibits a tarnish resistance such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance

10

## CLAIMS

1. A method for treating a finished or semi-finished shaped flatware, hollowware or jewellery article of a silver/germanium alloy that has a silver content of at least 77 wt % and a germanium content of between 0.4 and 7% the remainder principally being copper so as to reduce or further reduce tarnishing of the article such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance, said method comprising
- surface treating said article with ~~Use of an~~ alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide ~~for the surface treatment of an alloy of silver containing an amount of germanium that is effective to reduce firestain and/or tarnishing so as to reduce or further reduce tarnishing of the alloy such that a sample can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance.~~
2. The methoduse of claim 1, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide has C<sub>12</sub>-C<sub>24</sub> alkyl groups.
3. The methoduse of claim 1 or 2, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in an organic solvent.
4. The methoduse of claim 3, wherein the solvent containing the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is generally neutral.
5. The methoduse of claim 3 or 4, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in a solvent based on n-propyl bromide.

6. The methoduse of claim 3 or 4, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in (a) a composition obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in an organic solvent and adding to said solution a relatively concentrated aqueous soap or detergent, or (b) an aqueous dispersion obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in an organic solvent, adding to said solution a relatively concentrated aqueous soap or detergent, and diluting the resulting mixture with water.

7. The method of claim 1 or 2, wherein said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is in-or-(e) a composition obtainable by dissolving said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide direct in an aqueous mixture of an anionic surfactant and a neutral, -or amphoteric or zwitterionicnionic surfactant, said mixture being free from solvents other than water.

8. The method of claim 1 or 2, whrerin said article is treated with a water-based composition comprising said comprising said alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide and at least one of an amphoteric, nonionic or cationic surfactant in a concentration that is effective to solubilise the treatment agent.

<sup>8</sup>/<sub>9</sub>. The method of claim <sup>7</sup>/<sub>8</sub>, wherein said composition comprises as surfactant a betaine.

<sup>9</sup>/<sub>10</sub>. The method of claim <sup>8</sup>/<sub>9</sub>, wherein the betaine is cocamidopropyl betaine.

<sup>10</sup>/<sub>11</sub>. The method of any of claims <sup>7-9</sup>/<sub>7-9</sub>, further comprising an anionic surfactant.

<sup>11</sup>  
~~12.~~ The method of claim <sup>10</sup>~~11~~, wherein the anionic surfactant is of the formula  $RO-(CH_2CH_2)_nSO_3M$  wherein R represents  $C_{10}-C_{18}$  alkyl, n is 2-6 and M represents a monovalent cation.

5 <sup>12</sup>  
~~13.~~ The method of claim <sup>11</sup>~~12~~, wherein the anionic surfactant is a monovalent cation salt of laureth sulfate.

<sup>13</sup>  
~~14.~~ The composition of any of claims <sup>7 12</sup>~~8-13~~, comprising amphoteric or zwitterionic surfactant and anionic surfactant in a weight ratio of from 1:10 to 10:1.

~~7. The use of claim 6, wherein the combination comprises a treatment agent selected from an alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide, an anionic surface active agent and an amphoteric surface active agent in concentrations that are effective to solubilise the treatment agent.~~

<sup>14</sup>  
~~15.~~ The ~~method~~ use of claim <sup>6</sup>~~106~~, wherein the aqueous mixture comprises sodium laureth sulfate and cocamidopropyl betaine, the combination comprises an alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide, an anionic surface active agent and a neutral surface active agent in concentrations that are effective to solubilise the treatment agent.

~~9. The use of claim 1 or 2, wherein the alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide is contained in a polish or impregnated into a polishing cloth.~~

<sup>15</sup>  
~~106.~~ The ~~method~~ use of any preceding claim, wherein the alkanethiol or alkylthioglycollate is selected from stearyl mercaptan (octadecyl mercaptan), cetyl mercaptan (hexadecyl mercaptan), stearyl thioglycollate and cetyl thioglycollate.

~~11. The use of any preceding claim, for the surface treatment of an alloy that further comprises a grain refiner.~~

~~12. The use of any preceding claim for the surface treatment of a ternary alloy of silver, copper and germanium.~~

~~13. The use of claim 12, wherein the ternary alloy consists, apart from impurities and any grain refiner, of 80-96% silver, 0.1-5% germanium and 1-19.9% copper, by weight of the alloy.~~

<sup>14</sup>  
~~14.~~ The method of any preceding claim 12, wherein the ternary alloy consists, apart from impurities and grain refiner, of 92.5-98% silver, 0.3-3% germanium, and 1-7.2% copper, by weight of the alloy, together with 1-40 ppm boron as grain refiner.

<sup>15</sup>  
<sup>15</sup>  
~~15.~~ The method of claim 172, wherein the ternary alloy consists, apart from impurities and grain refiner, of 92.5-96% silver, 0.5-2% germanium, and 1-7% copper, by weight of the alloy, together with 1-40 ppm boron as grain refiner.

<sup>16</sup>  
~~16.~~ The method of any of claims 1-16, wherein the alloy is ~~a11~~ for the treatment of a quaternary alloy of silver, copper, zinc and germanium.

<sup>19</sup>  
<sup>20</sup>  
~~20.~~ The method of any preceding claim, comprising the further step of introducing the treated article into packaging.

<sup>21</sup>  
~~21.~~ The method of claim 20, wherein said packaging includes a presentation box.

<sup>22</sup>  
~~22.~~ The method of claim 21, wherein the packaging includes external wrapping for the presentation box.

17. ~~The use of claim 16, wherein the zinc is present in a ratio, by weight, to the copper of no more than 1:1.~~

5 18. ~~The use of any preceding claim, wherein the alloy is in the form of a finished or semi-finished article.~~

~~24~~

22 19. Use of a C<sub>12</sub>-C<sub>24</sub> alkanethiol, alkyl thioglycollate, dialkyl sulfide or dialkyl disulfide in the preparation of a tarnish inhibitor for an article of a  
10 silver/germanium alloy that has a silver content of at least 77 wt % and a germanium content of between 0.4 and 7%, the remainder principally being copper, so as to reduce tarnishing of the alloy such that a sample can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance.

15

22 20. A finished or semi-finished shaped flatware, hollowware or jewellery article of an alloy of silver, or a shaped article formed of said alloy, containing an amount of germanium that is effective to reduce firestain and/or tarnishing and that has been treated with a C<sub>12</sub>-C<sub>24</sub> alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide and that exhibits a tarnish resistance such that a sample of the alloy of which the article is made can be supported close above a 20% solution of ammonium polysulphide for at least 30 minutes while retaining a generally untarnished appearance.

25 21. ~~A method for manufacturing a tarnish-resistant silver article, which comprises the steps of:~~

~~forming a shaped article of an alloy of silver containing an amount of germanium that is effective to reduce firestain and/or tarnishing;~~

30 ~~surface treating the article with an alkanethiol, alkyl thioglycollate, dialkyl sulphide or dialkyl disulphide; and~~

~~introducing the article into packaging.~~



~~22. The method of claim 21, wherein said packaging includes a presentation box.~~

5 ~~23. The method of claim 22, wherein the packaging includes external wrapping for the presentation box.~~

The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ \_\_\_\_\_

# PCT

## CHAPTER II

### DEMAND

under Article 31 of the Patent Cooperation Treaty:  
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty.

For International Preliminary Examining Authority use only	
Identification of IPEA	Date of receipt of DEMAND
<b>Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION</b>	
Applicant's or agent's file reference MDX,004-PCT	
International application No. PCT/GB2004/002317	International filing date (day/month/year) 1 Jun 04
(Earliest) Priority date (day/month/year) 3 Jun 03	
Title of invention SILVER TERNARY ALLOY	
<b>Box No. II APPLICANT(S)</b>	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) MIDDLESEX SILVER CO. LIMITED Middlesex University Queensway Enfield Middlesex EN3 4SF United Kingdom	Telephone No.
	Facsimile No.
	Teleprinter No.
	Applicant's registration No. with the Office
State (that is, country) of nationality: GB	State (that is, country) of residence: GB
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Peter Gamon JOHNS 39 Richmond Drive Watford Hertfordshire WD1 3BQ United Kingdom	
State (that is, country) of nationality: GB	State (that is, country) of residence: GB
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	
State (that is, country) of nationality:	State (that is, country) of residence:
<input type="checkbox"/> Further applicants are indicated on a continuation sheet.	

**Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**The following person is ☐ agent ☐ common representativeand ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.Name and address: *(Family name followed by given name; for a legal entity, full official designation.  
The address must include postal code and name of country.)*

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☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.**Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION****Statement concerning amendments:\***

1. The applicant wishes the international preliminary examination to start on the basis of:

☐ the international application as originally filedthe description ☐ as originally filed☐ as amended under Article 34the claims ☐ as originally filed☐ as amended under Article 19 (together with any accompanying statement)☒ as amended under Article 34the drawings ☐ as originally filed☐ as amended under Article 342. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.3. ☒ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of the applicable time limit under Rule 69.1(d).4. ☐ The applicant expressly wishes the international preliminary examination to start earlier than at the expiration of the applicable time limit under Rule 54bis.1(a).

\* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: .....

☐ which is the language in which the international application was filed.☐ which is the language of a translation furnished for the purposes of international search.☐ which is the language of publication of the international application.☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.**Box No. V ELECTION OF STATES**

The filing of this demand constitutes the election of all Contracting States which are designated and are bound by Chapter II of the PCT.

**Box No. VI CHECK LIST**

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- |  |   |        |
|--|---|--------|
| 1. translation of international application                              | : | sheets |
| 2. amendments under Article 34   | : | sheets |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | sheets |
| 4. copy (or, where required, translation) of statement under Article 19  | : | sheets |
| 5. letter  | : | sheets |
| 6. other (specify)   | : | sheets |

For International Preliminary Examining Authority use only

received not received

<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- |  |  |
|--|--|
| 1. <input checked="" type="checkbox"/> fee calculation sheet                             | 5. <input type="checkbox"/> statement explaining lack of signature                         |
| 2. <input type="checkbox"/> original separate power of attorney                          | 6. <input type="checkbox"/> sequence listing in computer readable form                     |
| 3. <input type="checkbox"/> original general power of attorney                           | 7. <input type="checkbox"/> tables in computer readable form related to a sequence listing |
| 4. <input type="checkbox"/> copy of general power of attorney; reference number, if any: | 8. <input type="checkbox"/> other (specify):   |

**Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE**

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).

  
Paul Cole (Agent)

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1. Date of actual receipt of DEMAND:

2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):

3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.  
☐ The applicant has been informed accordingly.
4. ☐ The date of receipt of the demand is WITHIN the time limit of 19 months from the priority date as extended by virtue of Rule 80.5.
5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

6. ☐ The date of receipt of the demand is AFTER the expiration of the time limit under Rule 54bis.1(a) and item 7 or 8, below, does not apply.
7. ☐ The date of receipt of the demand is WITHIN the time limit under Rule 54bis.1(a) as extended by virtue of Rule 80.5.
8. ☐ Although the date of receipt of the demand is after the expiration of the time limit under Rule 54bis.1(a), the delay in arrival is EXCUSED pursuant to Rule 82.

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Demand received from IPEA on:

Accels

CHAPTER II

PCT

FEE CALCULATION SHEET

Annex to the Demand

<p>International application No. <b>PCT/GB2004/001373</b></p> <p>Applicant's or agent's file reference <b>MDX,001-PCT</b></p> <p>Applicant <b>MIDDLESEX SILVER CO. LIMITED</b></p>	<p>For International Preliminary Examining Authority use only</p> <p>Date stamp of the IPEA</p>								
<p><b>CALCULATION OF PRESCRIBED FEES</b></p> <p>1. Preliminary examination fee ..... <span style="float: right;">1530.00 <span style="border: 1px solid black; padding: 0 5px;">P</span></span></p> <p>2. Handling fee (<i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i>) ..... <span style="float: right;">129.00 <span style="border: 1px solid black; padding: 0 5px;">H</span></span></p> <p>3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box ..... <span style="float: right; border: 1px solid black; padding: 2px;">1659.00</span></p> <div style="text-align: right; border: 1px solid black; width: 150px; margin: 0 auto; padding: 2px;">TOTAL</div>									
<p><b>MODE OF PAYMENT</b></p> <table style="width: 100%;"> <tr> <td><input checked="" type="checkbox"/> authorization to charge deposit account with the IPEA (see below)</td> <td><input type="checkbox"/> cash</td> </tr> <tr> <td><input type="checkbox"/> cheque</td> <td><input type="checkbox"/> revenue stamps</td> </tr> <tr> <td><input type="checkbox"/> postal money order</td> <td><input type="checkbox"/> coupons</td> </tr> <tr> <td><input type="checkbox"/> bank draft</td> <td><input type="checkbox"/> other (specify):</td> </tr> </table>		<input checked="" type="checkbox"/> authorization to charge deposit account with the IPEA (see below)	<input type="checkbox"/> cash	<input type="checkbox"/> cheque	<input type="checkbox"/> revenue stamps	<input type="checkbox"/> postal money order	<input type="checkbox"/> coupons	<input type="checkbox"/> bank draft	<input type="checkbox"/> other (specify):
<input checked="" type="checkbox"/> authorization to charge deposit account with the IPEA (see below)	<input type="checkbox"/> cash								
<input type="checkbox"/> cheque	<input type="checkbox"/> revenue stamps								
<input type="checkbox"/> postal money order	<input type="checkbox"/> coupons								
<input type="checkbox"/> bank draft	<input type="checkbox"/> other (specify):								
<p><b>AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT</b> (This mode of payment may not be available at all IPEAs)</p> <p><input checked="" type="checkbox"/> Authorization to charge the total fees indicated above.</p> <p><input type="checkbox"/> (This check-box may be marked only if the conditions for deposit accounts of the IPEA so permit) Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.</p> <div style="text-align: right; margin-top: 10px;"> <p>IPEA/ <u>EP</u></p> <p>Deposit Account No.: <u>28050111</u></p> <p>Date: <u>13 Oct 04</u></p> <p>Name: <u>Paul Cole</u> <u>Brian Lucas</u></p> <p>Signature: <u>Brian Lucas</u></p> </div>									